There is a hole in my bucky

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SUPPLEMENTARY MATERIAL FOR

There is a Hole in my Bucky

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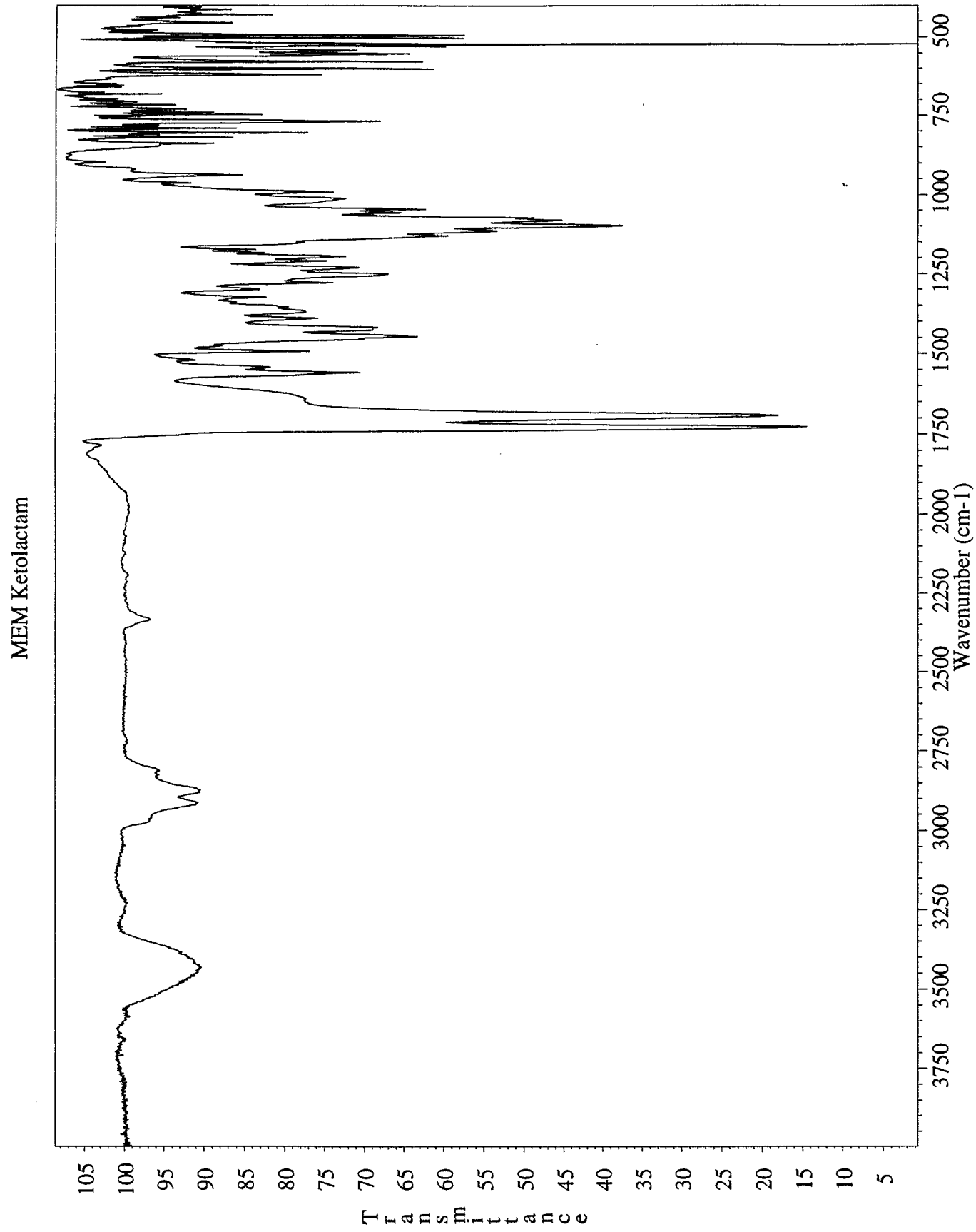
N-MEM- ketolactam 3 from 5.6 N-MEM-azafulleroid 1

A solution of 21 mg (25.5 μmole) 5.6 N-MEM-azafulleroid 1 in 75 ml HPLC grade 1,2-dichorobenzene was irradiated with a flood lamp provided with a sheet of Kapton 500HN (DuPont) as the light filter, while bubbling through a gentle stream of oxygen. The temperature of the reaction was maintained at 25-30°C using a water bath, cooled internally by tap water running through a copper spiral. After 3 hours, the whole reaction mixture was poured on a column of Silica/toluene (25 cm). Upon elution with toluene, a trace of starting material and the 1,2-dichlorobenzene were removed. Elution with toluene/EtOAc (9:1) gave the fraction containing pure ketolactam. The solvent was removed *in vacuo* , and the residue was dissolved in a minimum amount of CS_2. The product was precipitated with Et_2O (50 ml), the mixture was centrifuged, and the supernatant was decanted. The solid residue was washed with Et_2O (2 x 70 ml; suspended, centrifuged, decanted) and dried in a vacuum oven at 60°C overnight. Yield: 14 mg (64%).

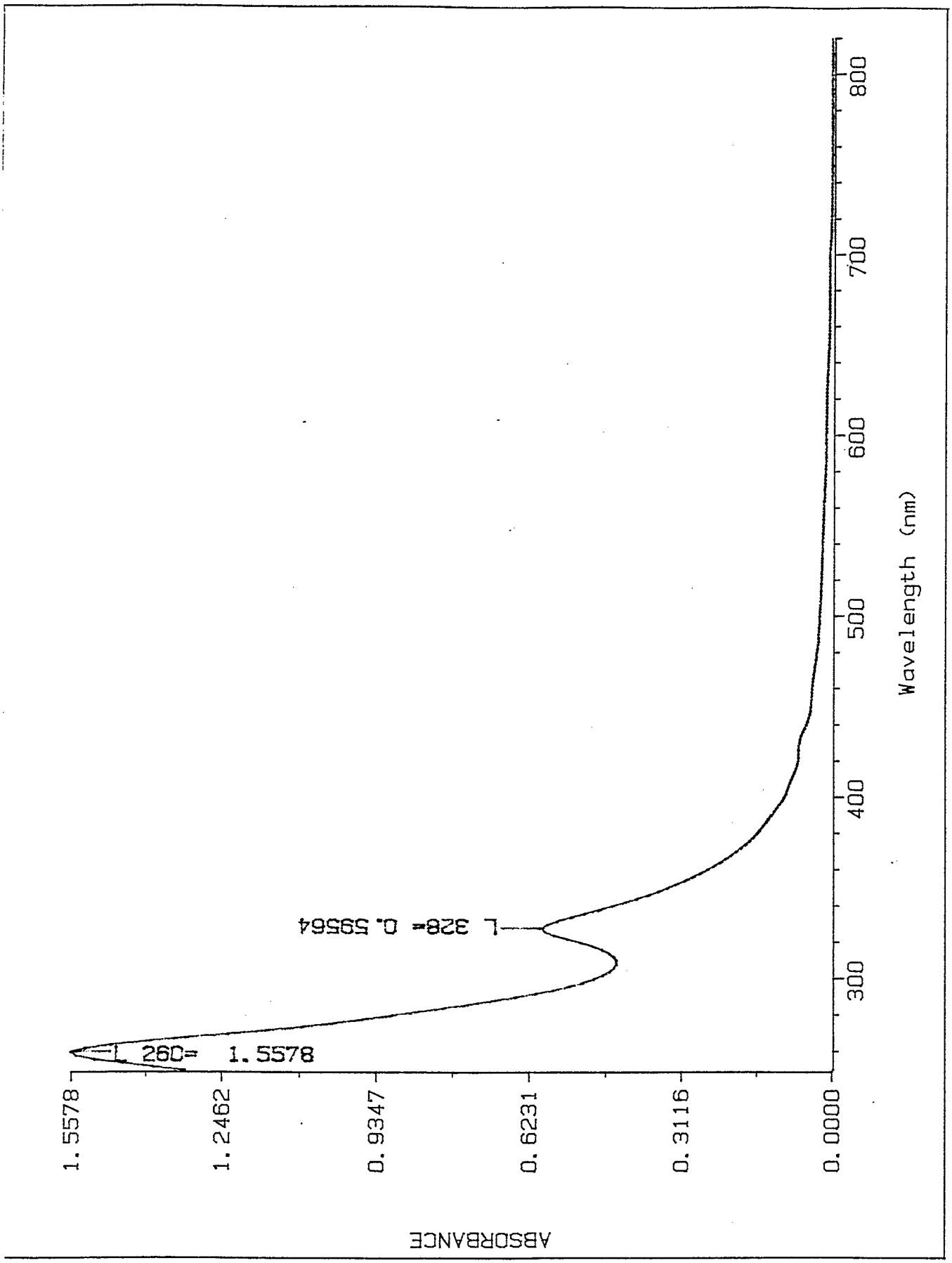
J7004-m2

^{1}H NMR (CS_2, TMS; 500 MHz, δ): 6.19, 5.83 (AB system; J_{AB} = 10.5 Hz; 2H; N-CH_2), 3.89 (m, 2H; O-CH_2CH_2), 3.48 (m, 2H; O-CH_2CH_2), 3.23 (s, 3H; OCH_3). ^{13}C NMR ($CDCl_3$, TMS; 125 MHz): 198.49, 163.58, 150.11, 149.40, 147.75, 147.43, 147.11, 146.74, 146.63, 146.43, 146.25, 146.13, 146.08, 145.93, 145.81, 145.46, 145.40, 145.34, 145.16, 144.91, 144.84, 144.55, 144.45, 144.25, 144.16, 143.99, 143.84, 143.78, 143.63, 143.52, 143.43, 142.69, 141.63, 141.22, 141.05, 140.41, 139.75, 139.61, 139.49, 139.28, 138.72, 137.93, 136.72, 136.03, 135.96, 135.71, 134.05, 133.96, 132.99, 132.43, 132.05, 128.23, 127.96, 80.61, 71.64, 70.04, 59.11. FTIR (KBr): 1727 s, 1693 s, 1083 s ,1075 s, 619 m, 600 m, 578 m, 559 w, 554 m, 552 m, 541 w, 528 m, 522 s, 503 m, 494 m, 458 m (cm^{-1}). UV-vis ($CHCl_3$) λ_{max} (nm): 260, 328, 428 (sh), 466 (sh), 616 (sh), 688. FABMS (NBA) *m/z*: 856 $(M+H)^+$. Anal. Calcd for $C_{64}H_9NO_4$: C, 89.82; H, 1.06; N, 1.64. Found: C, 87.21; H, 1.32; N, 1.61.

J7004-m3



J 7004-m4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0.0000
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400
500
600
700
800
Wavelength (nm)
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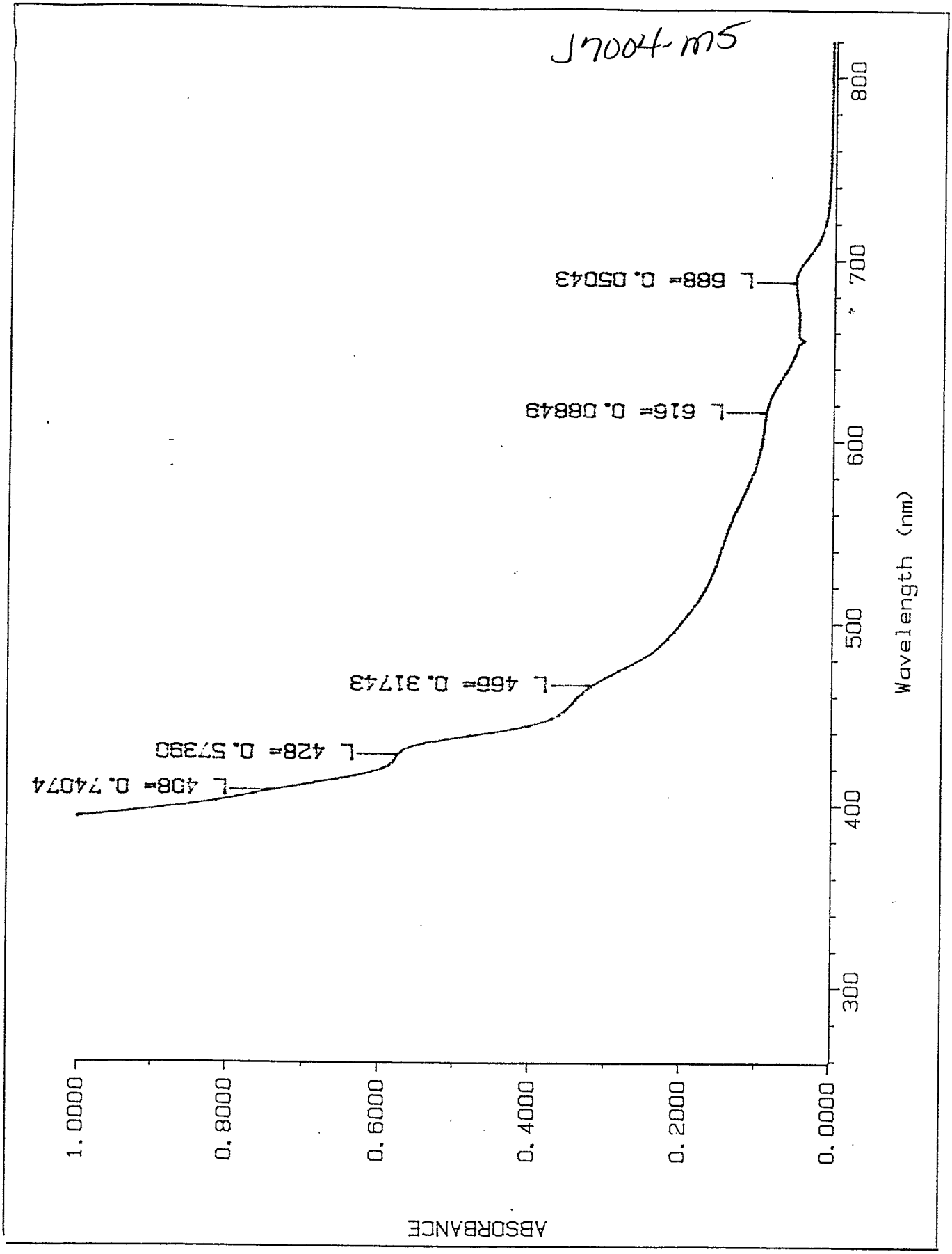
328= 0.59564


J7004-m5
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L 616= 0.08849
L 466= 0.31743
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L 408= 0.74074
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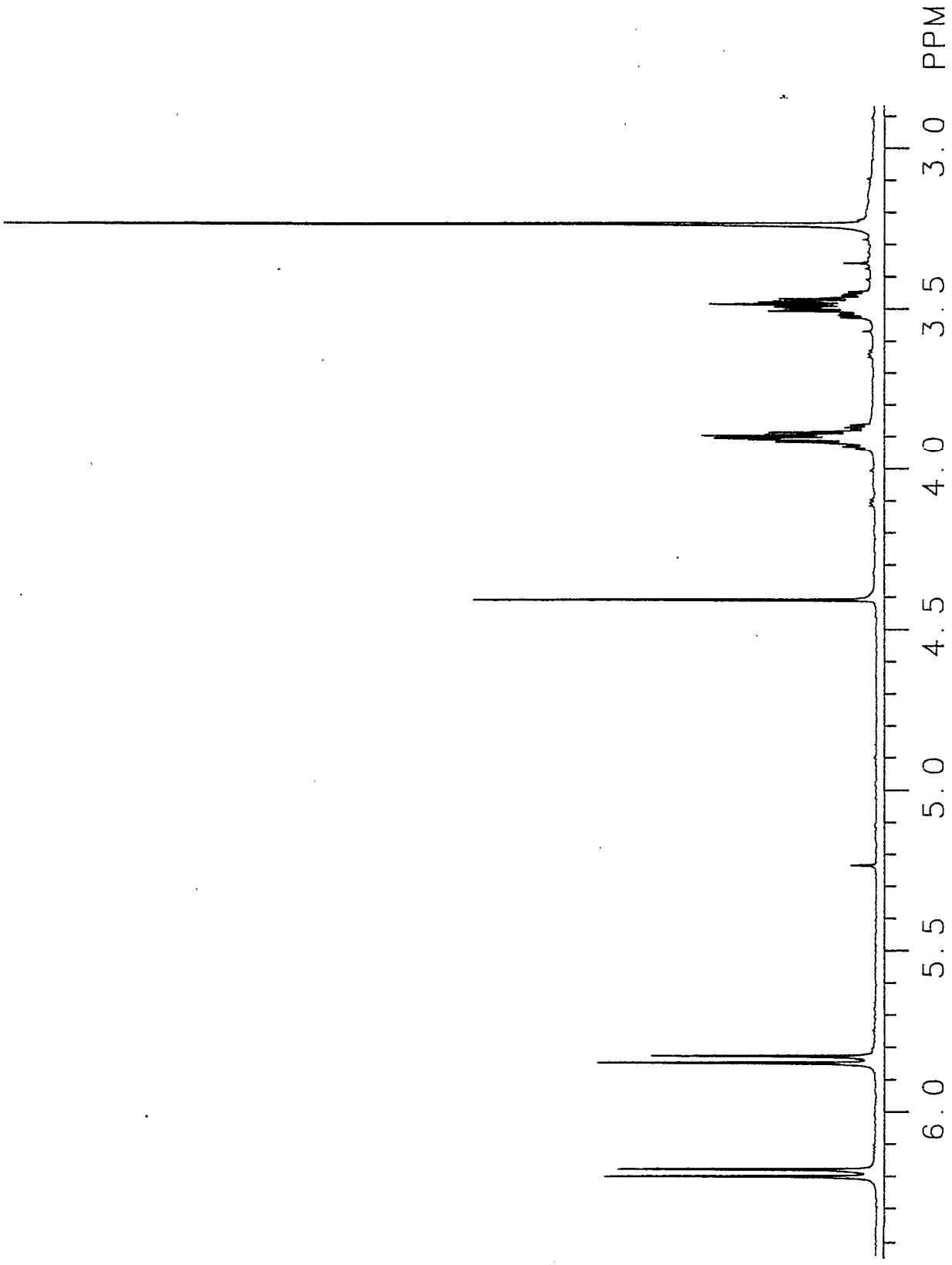
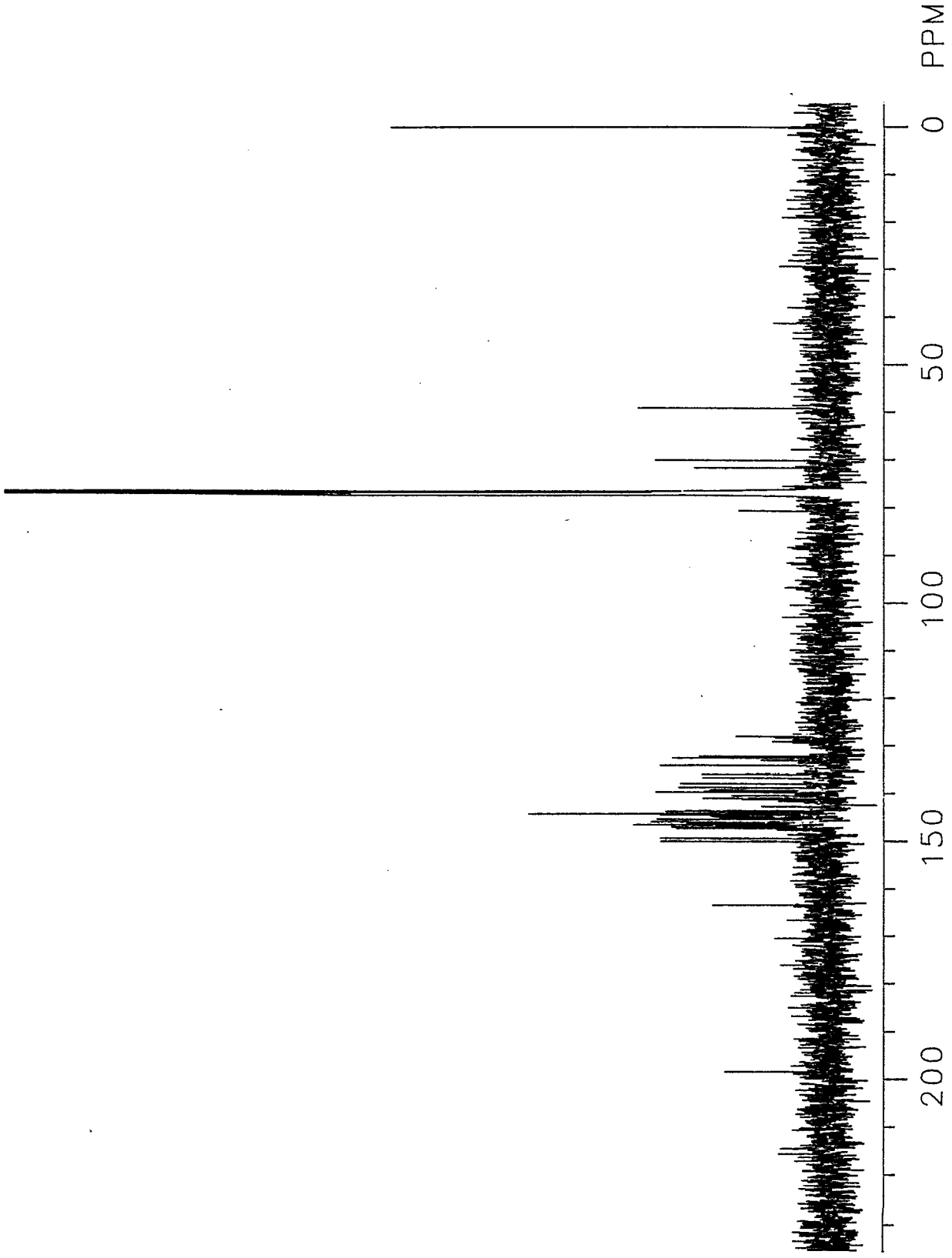
0.2000
0.0000
300
400
500
600
700
800
Wavelength (nm)

J7004-M6



J7004-m7

1-15-2-95



J7004-M8

2-15-2-95